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Tank Characterization Report for Single-Shell Tank 241-AX-104

Jim G. Field

Lockheed Martin Hanford Corp., Richland, WA 99352 U.S. Department of Energy Contract 8023764-9-K001

Lance C. Amato

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Abstract: This document summarizes the information on the historical uses, present status, and the sampling and analysis results of waste stored in Tank 241-AX-104. This report supports the requirements of the Tri-Party Agreement Milestone M-44-15C.

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Tank Characterization Report for Single-Shell Tank 241-AX-104

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2.0 RESPONSE TO TECHNICAL ISSUES

The following technical issue has been identified for tank 241-AX-104 (Brown et al. 1998).

• Tank 241-AX-104: What is the inventory and leachability of the waste in tank 241-AX-104 (Banning 1998)?

Additional technical issues required by Brown et al. (1997) and addressed by sampling events include:

- Safety screening: Does the waste pose or contribute to any recognized potential safety problems?
- Organic complexants: Does the possibility exist for a point source ignition in the waste followed by a propagation of the reaction in the solid/liquid phase of the waste?
- Organic solvents: Does an organic solvent pool exist that may cause a fire or ignition of organic solvents in entrained waste solids?

Data from the analysis of auger samples, tank headspace measurements, and tank vapor samples, along with available historical information, provided the means to respond to the technical issues. The following sections present the response. See Appendix B for sample and analysis data for tank 241-AX-104.

As described in Section B3.1, significant uncertainties exist regarding the representativeness of the riser 3A auger samples to the majority of the tank waste. To provide a radiologically conservative waste inventory, no means or confidence intervals were calculated using data from the riser 3A auger samples. However, because the riser 3A auger results do provide composition data for the waste under that particular riser, the results have been used in the safety screening assessment. Because analytical results from both risers 3A and 9G are used in the assessment, the safety screening data quality objective (DQO) requirement of two vertical waste profiles is considered to have been met.

2.1 TANK 241-AX-104 WASTE CHARACTERIZATION DATA QUALITY OBJECTIVE

Tank 241-AX-104 was selected as the preferred tank at which residual waste characterization could be conducted to support the Hanford Tanks Initiative (HTI) Project. The primary objective of the HTI Project is to provide a technical basis for the design and regulatory decisions for the waste retrieval and closure of high-level waste tanks at the Hanford Site. To meet the needs of the HTI Project, determination of the tank 241-AX-104 waste inventory was required, as well as a waste leachability study. Tank 241-AX-104 Waste Characterization Data Quality Objective (Banning 1998), referred to as the HTI DQO, was prepared to define the sampling and analytical requirements needed to resolve these issues.

Regarding waste inventory, the analytes of concern for the HTI Project are antimony, arsenic, barium, cadmium, chromium, lead, silver, nitrate, nitrite, ²⁴¹Am, ⁶⁰Co, ¹³⁷Cs, ^{239/240}Pu, ⁷⁹Se, ⁹⁰Sr,

and ⁹⁹Tc (Banning 1998). Appendix B provides a detailed description of the analytical results. The HTI DQO does not establish notification limits for the individual analytes.

Data were obtained for all required analytes (Esch 1998); however, the ⁷⁹Se data are considered suspect and should be used with caution. During the liquid scintillation analysis, energy was observed in the area where ⁷⁹Se would be expected. However, because no actual peak was observed, it is believed that the energy was not from ⁷⁹Se but was instead caused by interference from high levels of ¹³⁷Cs in the waste. Consequently, a mean for ⁷⁹Se was not derived.

Results from the required leach study are presented in Appendix B. Deviations were required from the original work plan. The leach test was to be performed on a composite of material from auger and light-duty utility arm samples. Because of delays in deploying the light-duty utility arm, the decision was made to proceed with the leach test on a composite of the auger samples only (Schreiber 1998c). A determination was also made to restrict the composite material to only the riser 9G augers because of concerns about the representativeness of the riser 3A auger samples. Results for all required analytes were obtained except ⁷⁹Se. As with the analyses on the individual auger samples, no notification limits are established by the HTI DQO.

2.2 SAFETY SCREENING

The data needed to screen the waste in tank 241-AX-104 for potential safety problems are documented in *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995). These potential safety problems are exothermic conditions in the waste, flammable gases in the waste and/or tank headspace, and criticality conditions in the waste. Each condition is addressed separately below.

2.2.1 Exothermic Conditions (Energetics)

The first requirement outlined in the safety screening DQO (Dukelow et al. 1995) is to ensure there are not sufficient exothermic constituents (organic or ferrocyanide) in tank 241-AX-104 to pose a safety hazard. The safety screening DQO required the waste sample profile be tested for energetics every 24 cm (9.5 in.) to determine whether the energetics exceeded the safety threshold limit. The threshold limit for energetics is 480 J/g on a dry weight basis.

The auger sampling and analysis plan (SAP) (Schreiber 1998a) required a differential scanning calorimetry (DSC) analysis to assess energetics. A DSC analysis was performed on the riser 3A augers. However, a DSC analysis could not be performed on the riser 9G samples because of the high dose rates associated with the auger samples. Consequently, Schreiber (1998b) directed that the DSC analysis be replaced by a total organic carbon (TOC) analysis by furnace oxidation. Because no ferrocyanide is expected in the tank based on the process history, TOC would be the source of any energetics. Therefore, a TOC analysis provides equivalent results to the DSC analysis.

A threshold limit of 45,000 μ g C/g (dry weight) has been established for TOC concentration (Adams 1998a). Upper limits (ULs) to 95 percent confidence intervals for the analytical sample means are used for comparison to the threshold. For the riser 9G samples, all TOC results were below detection levels, so no confidence intervals were calculated. After converting the riser 3A

screening is no longer an issue because headspace vapor (sniff) tests are required for the safety screening DQO (Dukelow et al. 1995), and the toxicity issue was closed for all tanks (Hewitt 1996).

2.5.2 Tank Waste Heat Load

A factor in assessing tank safety is the heat generation and temperature of the waste. Heat is generated in the tanks from radioactive decay. An estimate of the tank heat load based on the 1997 auger sampling event was derived using the radionuclide data, as shown in Table 2-1. Note that to provide the most conservative estimate, a density of 1.8 g/mL and a volume of 28.4 kL (7.5 kgal) were used when converting concentrations to inventories.

The best-basis inventory radionuclide data yielded a heat load estimate of 18,100 W (61,800 Btu/hr) (note that this value is biased high because the best-basis radionuclide inventories are decay corrected to January 1, 1994). This estimate is above the 11,700 W (40,000 Btu/hr) threshold that separates high- and low-heat-load tanks. Tank 241-AX-104 is not currently considered a high-heat-load tank (Hanlon 1998). Other heat load estimates of 2,960 W (10,100 Btu/hr) (based on process history [Agnew et al. 1997a]) and 4,220 W (14,400 Btu/hr) (based on tank headspace temperatures [Kummerer 1995]) indicate that the tank may not be a high-heat-load tank. Because of these conflicting heat load estimates and the uncertainty surrounding the waste volume, a definitive categorization regarding heat load cannot be made at this time.

Table 2-1. Projected Heat Load.

Analyte	Inventory (Ci) ¹	W/Ci	W
²⁴¹ Am	972	0.0328	31.9
⁶⁰ Co	334	0.0154	5.14
¹³⁷ Cs	63,300	0.00472	299
¹⁵⁴ Eu	1,870	0.00898	16.8
¹⁵⁵ Eu	1,700	7.23E-04	1.23
²³⁹ Pu	286	0.0305	8.72
²⁴⁰ Pu	54.5	0.0306	1.67
⁹⁰ Sr	2.64E+06	0.00670	17,700
Total			18,100

Note:

¹Best-basis inventory values

2.6 SUMMARY

The results of all analyses performed to address potential safety issues showed that primary analytes did not exceed safety decision threshold limits. The heat load categorization remains unresolved. A summary of the technical issues is presented in Table 2-2.

Table 2-2. Summary of Technical Issues.

Issue	Sub-issue	Result
Tank 241-AX-104	n/a	A waste inventory was derived for all required
waste inventory		analytes except ⁷⁹ Se based on the auger analytical
and leach study		results. Results from the leach study were obtained
	:	for all required analytes except ⁷⁹ Se.
Safety screening	Energetics	The riser 9G TOC results were below detection
		limits. No exotherms were observed for the
		riser 3A samples, and the dry-weight TOC results
		and 95 percent confidence interval ULs for these
		samples were at least eight times below the
		45,000 μg C/g limit.
	Flammable gas	Results from two separate combustible gas meter
		readings of the tank headspace were below the 25%
		LEL threshold (both 0% of the LEL).
	Criticality	All results and 95 percent confidence interval ULs
		for total alpha (riser 3A data) and ^{239/240} Pu (riser 9G
		data) were below 34.2 μCi/g.
Organic	Safety	No exothermic behavior was observed in the riser
complexants1	categorization (safe)	3A samples, and all TOC results and 95 percent
		confidence interval ULs were below 45,000 µg C/g.
Organic solvents	Solvent pool size	Organic pool size is estimated to be 0.03 m ² , well
	_	below the limit of 1 m ² .

Note:

¹The organic complexants safety issue was closed in December 1998 (Owendoff 1998).

4.0 RECOMMENDATIONS

The results of all analyses performed to address potential safety issues showed that the TOC concentration, headspace flammable gas concentration, and $^{239/240}$ Pu concentration were below their respective safety decision threshold limits. Vapor samples showed the estimated organic pool size was well below the safety limit of 1 m².

Table 4-1 summarizes the Project Hanford Management Contractor (PHMC) TWRS Program review status and acceptance of the sampling and analysis results reported in this TCR. All issues required to be addressed by sampling and analysis are listed in column 1 of Table 4-1. Column 2 indicates by "yes" or "no" whether issue requirements were met by the sampling and analysis performed. Column 3 indicates concurrence and acceptance by the program in PHMC/TWRS responsible for the applicable issue. A "yes" in column 3 indicates that no additional sampling or analyses are needed. Conversely, a "no" indicates additional sampling or analysis may be needed to satisfy issue requirements.

Sampling and analysis for the tank 241-AX-104 waste characterization DQO (Banning 1998) have been only partially performed; only one tank stratum, the tank floor, has been sampled to date. Future sampling of the waste on the remaining two tank strata (the tank walls/hardware and the tank ceiling) has been suspended (Sieracki 1999).

Results from the 1997 auger samples are considered adequate for assessing the issues of the safety screening DQO. As discussed in Section 2.0, data from the riser 3A auger samples were not included in derivation of tank means in order to provide the most radiologically conservative waste inventory. However, although likely different from a majority of the tank solids, the riser 3A augers do provide a profile of the waste underneath the riser. Consequently, the results were used in combination with the riser 9G data to perform the safety screening assessment. Use of two vertical profiles satisfies the sampling requirement of the safety screening DQO.

Table 4-1. Acceptance of Tank 241-AX-104 Sampling and Analysis.

Issue	Sampling and Analysis Performed	TWRS/PHMC Program Acceptance
Tank 241-AX-104 waste characterization DQO	Partial	Partial
Organic complexants memorandum of understanding ¹	Yes	Yes
Organic solvents DQO ²	Yes	Yes
Safety screening DQO	Yes	Yes

Notes:

¹The organic complexants safety issue was closed in December 1998.

²The organic solvents issue is expected to be closed in 1999.

Table 4-2 summarizes the status of PHMC TWRS Program review and acceptance of the evaluations and other characterization information contained in this report. Column 1 lists the different evaluations performed in this report. Column 2 shows whether issue evaluations have been completed or are in progress. Column 3 indicates concurrence and acceptance with the evaluation by the program in PHMC/TWRS that is responsible for the applicable issue. A "yes" indicates that the evaluation is completed and meets all issue requirements.

The evaluation for the Tank 241-AX-104 Waste Characterization DQO (Banning 1998) can only partially be completed as a result of suspension of HTI work activities (Sieracki 1999).

Table 4-2. Acceptance of Evaluation of Characterization Data and Information for Tank 241-AX-104.

Issue	Evaluation Performed	TWRS/PHMC Program Acceptance
Tank 241-AX-104 waste caracterization DQO	Partial	Partial
Organic complexants memorandum of understanding ¹	Yes	Yes
Organic solvents DQO ²	Yes	Yes
Safety screening DQO	Yes	Yes

Notes:

¹The organic complexants safety issue was closed in December 1998.

²The organic solvents issue is expected to be closed in 1999.

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Table B2-1. Integrated Data Quality Objective Requirements for Tank 241-AX-104. (2 sheets)

Sampling Event	Applicable DQOs	Sampling Requirements	Analytical Requirements
Vapor sampling ⁴	Hazardous vapor Osborne and Buckley (1995) Organic solvents Meacham et al. (1997) ⁵	SUMMA™ canisters, triple sorbent traps, sorbent tube trains	Flammable gas, organic vapors, permanent gases, total nonmethane hydrocarbons

Notes:

B2.1 DESCRIPTION OF 1997 AUGER SAMPLING EVENT

The intent of the 1997 auger sampling was to obtain two vertical profiles of the tank waste. Vertical profiles are needed to satisfy the safety screening DQO (Dukelow et al. 1995) and the organic complexant memorandum of understanding (Schreiber 1997). Safety screening analyses include: total alpha activity to determine criticality, DSC to ascertain the fuel energy value, and thermogravimetric analysis (TGA) to obtain the total moisture content. In addition, combustible gas meter readings in the tank headspace are performed to measure tank headspace flammability. The safety screening DQO also requires bulk density measurements for use in calculations. The organic complexant MOU requires DSC and TGA. Both documents list TOC (by persulfate oxidation) as a secondary analyte.

The 1997 auger sampling only partially satisfied the requirements of the HTI DQO (Banning 1998). This DQO strives to evaluate the spatial variance in analyte concentration within the tank waste. To meet this objective, the interior of the tank has been divided into three strata for sample collection. The three strata are the floor, walls/hardware, and tank dome. Only the floor stratum can be sampled using the auger sampling method. Sampling of the remaining two strata using the light duty utility arm (LDUA) has been suspended (Sieracki 1999). The analyses required by the HTI DQO include ICP, ion chromatography (IC), liquid scintillation for ⁹⁹Tc and

¹Schreiber (1998a)

²TOC is a secondary analyte for both the safety screening and organic complexant DQOs.

³The leach tests are performed in accordance with Schreiber (1998b).

⁴Buckley (1997)

⁵The vapor sampling occurred before the release of the organic solvents DQO. The requirements of this DQO have retroactively been applied to the January 1997 data.

⁷⁹Se, gamma energy analysis (GEA) for ¹³⁷Cs, ⁶⁰Co, and ²⁴¹Am, alpha counting for ^{239/240}Pu and ²⁴¹Am, beta counting for ⁹⁰Sr, an ICP/mass spectrometry (MS) screen for major fission products, and leach tests. The leach tests were to be performed on a composite of the floor stratum material from the auger and LDUA samples. The leach tests include ICP for seven metals, IC for nitrate and nitrite, ⁹⁹Tc by ICP/MS and liquid scintillation, ⁷⁹Se by liquid scintillation, ¹³⁷Cs and ⁶⁰Co by GEA, total alpha and total beta counting, and pH. To provide a baseline for the analyte concentrations, all of the same analyses except pH were to be performed directly on the composite.

Four auger samples were removed from tank 241-AX-104 in November 1997, two each from risers 3A and 9G. The auger samples obtained through riser 3A were taken on November 13, while those removed from riser 9G were taken on November 14 and 21. Sampling was performed in accordance with the auger sampling and analysis plan (SAP) (Schreiber 1998a). Sampling was performed using ten-inch auger samplers. Lithium bromide solution was not added to the drill string during sampling. The chain-of-custody forms for the riser 9G auger samples noted that high levels of contamination were detected under the lids of the shipping casks. A combustible gas meter reading was taken in the tank headspace before sampling. Further discussion of this measurement is provided in Section B2.2.

B2.1.1 Sample Handling

The four auger samples were shipped to the 222-S Laboratory, where they were extruded and photographed. Table B2-2 presents the extrusion information and sample descriptions. No drainable liquid was collected from any of the segments.

Table B2-2.	Extrusion	Information	and Sa	ample :	Descriptions.	(2 sheets)
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Riser	Auger Sample	Weight (g)	Auger Number	Sample Description
3A	97-AUG-001	96.8	Whole	Solids were collected from flutes 1 through 12 as well as the auger liner. Sample appeared as a mixture of fine, light brown powder and darker, coarser material. There were two small clear pieces of plastic or glass that were not collected with the sample.
	97-AUG-002	39.5	Whole	Solids were collected from flutes 1 through 11 as well as the auger liner. Sample appeared as a mixture of fine, light brown powder and darker, coarser material.

recorded. Within one hour after stirring had ceased, the mixture of solids and liquids had separated into three fractions. The sample appeared to have heavier sludge in the bottom, clear liquid on top of the sludge, and a less dense layer of solids floating on the liquid (Crawford 1998).

Because of problems with unreliable stirring, the magnetic stir bar was replaced with a paddle stirrer that was mounted on the leach container lid. During conversion to the paddle stirrer, 36.03 g of solids and liquids were lost. To determine the amount of each phase that was lost, the residual wet solids remaining after the leach test were dried. The amount of water measured in the wet solids was 61.8 weight percent. Therefore, of the 36.03 g lost, 13.76 g were solids and 22.27 g were water. The resulting water to solids mixture based on this loss was 160.99 g water to 74.06 g solids (a 1:2.2 ratio of soilds to liquids) (Crawford 1998).

A sample was removed for analysis (this sample is the 24-hour or 1-day sample). Samples were also removed for analysis after 7 days, 30 days, and 90 days. The temperature and pH were measured at the time of each sampling. Additional temperature measurements were made each week.

B2.1.2 Sample Analysis

The 1997 auger samples were analyzed based on safety screening, organic complexant, and HTI issues. Tank 241-AX-104 Auger Sampling and Analysis Plan (Schreiber 1998a) and Tank 241-AX-104 Light Duty Utility Arm Sampling and Analysis Plan (Schreiber 1998b) directed the analysis.

B2.1.2.1 Auger Sampling and Analysis Plan. The suite of analyses specified in the auger SAP (Schreiber 1998a) included alpha counting for total alpha activity, ^{239/240}Pu, and ²⁴¹Am, DSC for energetics, TGA for water content, gravimetry for bulk density, IC for selected anions (bromide, nitrate, and nitrite), ICP/atomic emission spectroscopy (AES) for selected metals (aluminum, antimony, arsenic, barium, cadmium, chromium, iron, lead, lithium, manganese, nickel, silicon, silver, sodium, and uranium), GEA for ¹³⁷Cs and ⁶⁰Co, liquid scintillation for ⁹⁹Tc and ⁷⁹Se, and beta counting for ⁹⁰Sr. A screen for major fission products using ICP/MS was also requested.

Several deviations to the auger SAP (Schreiber 1998a) were necessary because of the dry, powdery nature of the samples and the high concentrations of ⁹⁰Sr in the two augers from riser 9G (97-AUG-003 and 97-AUG-004). Homogenizing the dry samples generated a fine powder that easily became airborne, increasing the risk for contamination spread. As a result, the samples were not handled outside the hot cell in their dry state. The acid and water digestions were started in the hot cell, and the fusion digestion was performed entirely in the hot cell.

Problems were encountered during completion of the acid digestion outside the hot cell. After receiving the acid-diluted sample from the hot cell, the technician attempted to transfer the entire sample to digestion beakers. Despite repeated rinses, a complete transfer could not be accomplished because the samples appeared to have "clumped" and adhered to the bottom of the sample vials. Because of the high dose rate of the samples, no exceptional efforts were made to recover the remaining material. Because of the difficulties in handling these samples, and concern over radiation exposure for the individual performing the digestion, no redigestion was requested (Esch 1998).

Any direct analyses that could be performed in the hot cell were done so (Schreiber 1998c). The TGA requirement was replaced by gravimetry because a gravimetric analysis can be performed in the hot cell. Differential scanning calorimetry is a direct method and cannot be done in a hot cell. Because of as low as reasonably achievable (ALARA) concerns caused by the substantial amount of radioactivity in the two auger samples from riser 9G, the DSC analysis was deleted from the analytical suite for these two samples. Instead, TOC analysis by furnace oxidation was performed. This method provides energetics data reasonably equivalent to that obtained by DSC, and reduces the risk to laboratory staff by using water digested samples rather than direct samples (Schreiber 1998c).

Schreiber (1998c) also directed that the analysis for total alpha activity be removed from the suite of analyses for the auger samples from riser 9G. Total alpha activity is used as a screening tool for criticality concerns. For this determination, it is assumed that all alpha activity originates from ²³⁹Pu. Because the auger samples were already being analyzed for ²³⁹Pu as required by the auger SAP (Schreiber 1998a), a total alpha analysis was unnecessary. Note that total alpha activity data is available for auger samples 97-AUG-001 and 97-AUG-002 because these samples had already been analyzed by the time the change was made.

Another deviation from the SAP concerned the density measurements. In an effort to conserve sample material, bulk density was not determined on any of the samples.

Although only specific metals and anions were requested during the respective ICP and IC analyses, results for many other metals and anions were obtained. These results are reported on an "opportunistic" basis, and are not subject to quality control (QC) requirements.

B2.1.2.2 Light-Duty Utility Arm Sampling and Analysis Plan. The LDUA SAP (Schreiber 1998b) directed analyses on three sample types: the whole LDUA sample; a composite of the auger and LDUA samples; and a leach test sample. However, as of February 1999, sampling using the LDUA system has been suspended (Sieracki 1999). Because the composite and leach test analytical data were needed to support other HTI project work, *Revision to Tank 241-AX-104 Leach Test Requirements* (Schreiber 1998d) directed the 222-S Laboratory to perform the analyses specified in the LDUA SAP on the auger samples.

The analytical suites for the composite and leach test samples were nearly identical. Each required alpha counting for total alpha activity, IC for selected anions (bromide, nitrate, and nitrite), ICP/AES for selected metals (antimony, arsenic, barium, cadmium, chromium, lead, lithium, and silver), GEA for ¹³⁷Cs and ⁶⁰Co, liquid scintillation for ⁹⁹Tc and ⁷⁹Se, and beta counting for total beta activity. In addition, the LDUA SAP specified a TGA analysis on the composite sample and a pH determination on the leach test sample. As discussed previously, a TGA analysis was not possible because of ALARA concerns. Data for "opportunistic" analytes were obtained during the ICP and IC analyses.

Higher than expected nitrate and nitrite concentrations were reported for the sample and duplicate from the composited solids. The nitrate concentration was approximately 10 times higher than the estimated values based on the auger results. However, the nitrate concentration of the composited auger samples appears to balance with the total cation inventory in the solids (Crawford 1998).

After obtaining results from the composite for ⁷⁹Se, the requirements of the LDUA SAP were modified to remove this analysis on the leach test sample. Further discussion on the logic behind this decision is provided in Section B2.1.3.10.

Analyses required by both the auger and LDUA SAPs were either performed directly on the solids or after digestion using water, acid, or fusion. Note that the fusion digestion for ⁹⁰Sr was repeated on the individual auger samples because a high concentration of the analyte was . detected in the preparation blank on the first preparation. The leach test analyses were performed directly on the liquid samples. All reported analyses were performed following the approved laboratory procedures given in Table B2-3. Tables B2-4 and B2-5 summarize the auger numbers, sample numbers, and analyses performed on each sample.

Table B2-3.	Analytical	Procedures	(2 sheets)
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Analysis	Method	Procedure Number		
Energetics	DSC	LA-514-114		
Percent water	TGA	LA-564-101		
Total alpha activity	Alpha counting	LA-508-101		
Flammable gas	Combustible gas analysis	WHC-IP-0030		
		IH 1.4 and IH-2.12		
TOC	Furnace oxidation	LA-344-105		
Metals	ICP/AES	LA-505-161		
Anions	IC	LA-533-105		
¹³⁷ Cs, ⁶⁰ Co	GEA	LA-548-121		
^{239/240} Pu, ²⁴¹ Am	AEA	LA-953-104		

Table B2-3. Analytical Procedures. (2 sheets)

Analysis	Method	Procedure Number		
⁹⁰ Sr	Beta counting	LA-220-101		
⁷⁹ Se	Liquid scintillation	LA-365-132		
⁹⁹ Tc	Liquid scintillation	LA-438-101		
Screen for major ICP/MS fission products		LA-506-101		
Total beta activity Beta counting		LA-508-101		
PH pH meter		LA-212-106		
Homogenization verification	LA/MS	LT-506-102		

Notes:

¹Schreiber (1998a and 1998b)

²WHC (1992)

Table B2-4. Sample Analysis Summary for Whole Samples. (2 sheets)

Riser	Auger Number	Sample Number	Preparation Method	Analyses
3A	97-AUG-001	S97T002280	Direct	Percent water (gravimetry)
		S97T002284	Direct	DSC
		S97T002288	Fusion	⁹⁰ Sr, AEA
97-AUG		S97T002301	Acid	⁹⁹ Tc and ⁷⁹ Se, ⁹⁰ Sr, AEA, ICP, GEA, total alpha, fission product screening
		S97T002305	Water	IC, TOC
		S98T001174	Fusion	⁹⁰ Sr
	97-AUG-002	S97T002281	Direct	Percent water (gravimetry)
		S97T002285	Direct	DSC
		S97T002289	Fusion	⁹⁰ Sr, AEA
		S97T002302	Acid	⁹⁹ Tc and ⁷⁹ Se, ⁹⁰ Sr, AEA, ICP, GEA, total alpha, fission product screening
		S97T002306	Water	IC, TOC
		S98T001175	Fusion	⁹⁰ Sr

(5 in.) Using video data, Reich (1997) estimated the debris mound under riser 3A to be 7.6 (3 in.) thick. The data suggest that of the 13- to 15-cm (5- to 6-in.) waste depth under riser 3A, only approximately the bottom 5 cm (2 in.) would be representative of the entire waste profile. Because the stroke lengths for 97-AUG-001 and 97-AUG-002 were 7.6 (3 in.) and 9.8 (3 % in.), respectively, the recovered material on the augers was likely composed of debris and therefore unrepresentative of the majority of the tank waste.

The in-tank measurements for riser 9G were more consistent. The magnetometer measurements yielded waste thickness readings between 4.3 and 12 cm (1.7 and 4.9 in.). The video data supported the magnetometer measurements, as a large waste mass is visible near the location of riser 9G. The video shows that riser 9G is on the edge of the mass, which would explain the different thickness readings. The temperature and radiation probe data also supported/confirmed the magnetometer waste thickness measurements (Reich 1997). Although an estimate of thickness for the debris mound under riser 9G was not made in Reich (1997), the mound would be expected to be smaller than the one under riser 3A. Riser 9G is only 15 cm (6 in.) in diameter and is located along the edge of the tank (see Appendix A), which would have reduced airflow. Only three airlift circulators are in the immediate vicinity of the riser, all on the right side. In contrast, riser 3A is 41 cm (16 in.) in diameter and is located near the center of the tank, almost in the middle of the two concentric rings of airlift circulators. Consequently, riser 3A would have been exposed to more aerosolized waste, and exposure would have come from all directions.

A portion of the debris under riser 3A is also believed to be tank corrosion products. Riser 3A has been used frequently in the past for gaining access to the tank waste. When risers are opened, debris or rust from the riser have been found to fall into the tank waste. Usually this is not a concern because of the large amount of waste and the small amount of rust. However, because tank 241-AX-104 does not contain much waste, a small amount of rust could potentially bias the analytical results. This was likely the case with the riser 3A samples, as demonstrated in the iron results. Auger samples 97-AUG-001 and 97 AUG-002 had mean iron results of 486,000 and 465,000 μ g/g, respectively. These results are the highest iron values recorded for any tank on the Hanford Site. Except for one 202,000 μ g/g result for tank 241-AW-106, the riser 3A iron results are nearly four times the results obtained on any other tank. The waste type in tank 241-AX-104 (PUREX high-level waste) is expected to have a high iron concentration, although the Agnew et al. (1997) estimate of 123,000 μ g/g is still nearly four times below the riser 3A auger results. Riser 9G had seen limited use before the 1997 auger sampling. The riser 9G samples had means of 265,000 and 277,000 μ g/g – more reasonable but also possibly showing a high bias as a result of some contamination by corrosion products.

Historical tank waste temperatures, which have averaged 36 °C (96 °F) since 1976, indicate that the waste should contain substantial amounts of radioactivity. The tank temperature data implies that a majority of the tank waste may be composed of the material sampled through riser 9G. If the waste were solely composed of the riser 9G material, the waste temperatures could be even higher; however, several of the parameters governing the thermal response of the tank (e.g., convective heat transfer) are not well defined. Section 2.5.2 presented a comparison of heat

loads based on radionuclide analytical data (using riser 9G results only) and tank waste temperatures. The analytical data-based heat load was approximately four times that derived from waste temperatures (Kummerer 1995).

Based on the available information, it is obvious that the material from risers 3A and 9G are substantially different, and that the riser 9G material is more closely related to the P2 waste type expected to be in the tank. Unfortunately, it is not known what fractions of waste the riser 3A samples and the riser 9G samples represent. For the purpose of deriving tank composition and inventory estimates, it was assumed that the fraction of waste represented by the riser 3A samples was minor compared to the P2 waste represented by the riser 9G samples; this assumption is principally based on the temperature data from the tank. Consequently, no data from the riser 3A samples were used in determining means and inventory estimates. Calculating means and inventories in this manner provides the most radiologically conservative estimates. However, omitting the riser 3A sample data may bias the estimates.

B3.2 QUALITY CONTROL ASSESSMENT

The usual QC assessment includes an evaluation of the appropriate standard recoveries, spike recoveries, duplicate analyses, and blanks that are performed in conjunction with the chemical analyses. All pertinent QC tests were conducted on the 1997 auger samples, allowing a full assessment regarding the accuracy and precision of the data. The auger and LDUA SAPs (Schreiber 1998a and 1998b, respectively) established specific criteria for all analytes. Sample and duplicate pairs with one or more QC results outside the specified criteria were identified by footnotes in the data summary tables. Because the opportunistic analytes were not required by either SAP and therefore do not have defined QC parameters, a quality control assessment was not performed on the opportunistic data.

The standard and spike recovery results provide an estimate of analysis accuracy. If a standard or spike recovery is above or below the given criterion, the analytical results may be biased high or low, respectively. Nearly all standard recoveries were within the required limits. Two ¹³⁷Cs standard recoveries, one ⁹⁰Sr standard recovery, and one ⁹⁹Tc standard recovery were slightly above the limit.

Matrix spike recoveries may have been affected for some analytes because of the incomplete transfer of sample material during the acid digestion. Spike recovery failures were noted for silicon, silver, iron, sodium, and uranium during the ICP analysis. The silicon failure may be attributed to "noise" near the detection limit, since most of the sample results were less than five times the detection limit. However, the subsamples had acid added to them before they were loaded out the hot cell, and sat in a vial for a longer time than usual before digestion. Leaching of silicon from the borosilicate glass may have occurred at this time as well as during the acid

- Crawford, B. A., 1998, *Tank 241- AX-104 Residual Solids Leach Test Results*, TWR-3548, Rev. 0, Numatec Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
- Dukelow, G. T., J. W. Hunt, H. Babad, and J. E Meacham, 1995, *Tank Safety Screening Data Quality Objective*, WHC-SD-WM-SP-004, Rev. 2, Westinghouse Hanford Company, Richland, Washington.
- Esch, R. A., 1998, Tank 241-AX-104, Auger Samples, 97-AUG-001, 97-AUG-002, 97-AUG-003 and 97-AUG-004 Analytical Results for the Final Report, HNF-SD-WM-DP-298, Rev. 0, Waste Management Federal Services of Hanford, Inc. for Fluor Daniel Hanford, Inc., Richland, Washington.
- Kummerer, M., 1995, *Heat Removal Characteristics of Waste Storage Tanks*, WHC-SD-WM-SARR-010, Rev. 1, Westinghouse Hanford Company, Richland, Washington.
- Lockrem, L. L., 1997, Revised Data Tables for Tank Vapor Database on Tanks 241-A-106, 241-AX-104, and 241-TX-106, (letter NHC-9756182 to M. R. Adams, July 17), Numatec Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
- Meacham, J. E., D. L. Banning, M. R. Allen, and L. D. Muhlestein, 1997, *Data Quality Objective to Support Resolution of the Organic Solvent Safety Issue*, HNF-SD-WM-DQO-026, Rev. 0, Duke Engineering and Services Hanford, Inc. for Fluor Daniel Hanford, Inc., Richland, Washington.
- Osborne, J. W., and L. L. Buckley, 1995, *Data Quality Objectives for Tank Hazardous Vapor Safety Screening*, WHC-SD-WM-DQO-002, Rev. 2, Westinghouse Hanford Company, Richland, Washington.
- Reich, F. R., 1997, 241-AX-104 Residual Waste Volume Estimate, HNF-SD-HTI-ER-001, Rev. 0, SGN Eurisys Services Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
- Schreiber, R. D., 1998a, *Tank 241-AX-104 Auger Sampling and Analysis Plan*, HNF-SD-WM-TSAP-149, Rev. 0A, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
- Schreiber, R. D., 1998b, *Tank 241-AX-104 Light Duty Utility Arm Sampling and Analysis Plan*, HNF-2071, Rev. 0, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Schreiber, R. D., 1998c, Revision to Tank 241-AX-104 Analytical Requirements Because of Sample Handling Difficulties, (internal memorandum 7A110-98-005 to R. A. Esch and D. B. Hardy, March 18), Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
- Schreiber, R. D., 1998d, "Revision to Tank 241-AX-104 Leach Test Requirements," (internal memorandum 7A110-98-014 to B. A. Crawford and R. A. Esch, May 21), Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
- Schreiber, R. D., 1997, Memorandum of Understanding for the Organic Complexant Safety Issue Data Requirements, HNF-SD-WM-RD-060, Rev. 0, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
- Sieracki, S. A., 1999, Contract No. DE-AC06-96RL13200 Suspension of Hanford Tank
 Initiative (HTI) Project Work Activities, (letter 9951315A to R. D. Hanson, February 25),
 U.S. Department of Energy, Richland Operations Office, Richland, Washington.
- Starr, J. L., 1977, 104-AX Sludge Analysis, (letter to F. M. Jungfleisch, November 2), Rockwell International, Richland, Washington.
- Viswanath, R. S., G. S. Caprio, J. G. Douglas, M. J. Duchsherer, E. S. Mast, L. A. Pingel, M. Stauffer, D. B. Bonfoey, and G. A. Fies, 1997, Tank Vapor Sampling and Analysis Data Package for Tank 241-AX-104, Sampled January 23, 1997, Rev. 0C, Numatec Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
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- Brown, T. M., J. W. Hunt, and L. J. Fergestrom, 1997, *Tank Characterization Technical Sampling Basis*, HNF-SD-WM-TA-164, Rev. 3, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Summarizes the 1997 technical basis for characterizing tank waste and assigns a priority number to each tank.
- Brown, T. M., J. W. Hunt, and L. J. Fergestrom, 1998, *Tank Characterization Technical Sampling Basis*, HNF-SD-WM-TA-164, Rev. 4, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Summarizes the 1998 technical basis for characterizing tank waste and assigns a priority number to each tank.
- Buckley, L. L., 1997, Vapor Sampling and Analysis Plan, HNF-SD-WM-TSAP-126, Rev. 0, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Contains vapor sampling and analysis procedure for 200 Area Tanks.
- Crawford, B. A., 1998, *Tank 241-AX-104 Residual Solids Leach Tests*, HNF-SD-HTI-TP-001, Rev. 1, Numatec Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Describes the test plan for the leach test on the composited waste from the 1997 auger sampling event.
- DOE-RL, 1996, Recommendation 93-5 Implementation Plan, DOE/RL-94-0001, Rev. 1, U.S. Department of Energy, Richland, Washington.
 - Describes the organic solvents issue and other tank issues.

- Hall, K. M., 1998, Extension of Tank 241-AX-104 Format III Report Deadline,
 (internal memorandum 7A120-98-003 to R. A. Esch, K. L. Powell, and
 C. M. Seidel, January 19), Lockheed Martin Hanford Corp. for
 Fluor Daniel Hanford, Inc., Richland, Washington.
 - Extended the Format III report deadline because of delays in sample homogenization and subsampling of the 1997 auger samples.
- Schreiber, R. D., 1998, *Tank 241-AX-104 Auger Sampling and Analysis Plan*, HNF-SD-WM-TSAP-149, Rev. 0A, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Contains sampling and analysis requirements for tank 241-AX-104 based on applicable DQOs.
- Schreiber, R. D., 1998, Tank 241-AX-104 Light Duty Utility Arm Sampling and Analysis Plan, HNF-2071, Rev. 0, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Contains requirements for collecting and analyzing samples from tank 241-AX-104 using the light duty utility arm.
- Schreiber, R. D., 1998, Revision to Tank 241-AX-104 Analytical Requirements Because of Sample Handling Difficulties, (internal memorandum 7A110-98-005 to R. A. Esch and D. B. Hardy, March 18), Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Contains changes made to the analytical plan for the 1997 auger samples because of sample handling difficulties.
- Schreiber, R. D., 1998, "Revision to Tank 241-AX-104 Leach Test Requirements," (internal memorandum 7A110-98-014 to B. A. Crawford and R. A. Esch, May 21), Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Contains changes made to the leach test analytical plan for the 1997 auger samples because of representativeness concerns for the riser 3A samples.

Sieracki, S. A., 1999, Contract No. DE-AC06-96RL13200 – Suspension of Hanford Tank Initiative (HTI) Project Work Activities, (letter 9951315A to R. D. Hanson, February 25), U.S. Department of Energy, Richland Operations Office, Richland, Washington.

Ie. Data Quality Objectives and Customers of Characterization Data

- Banning, D. L., 1998, *Tank 241-AX-104 Waste Characterization Data Quality Objective*, HNF-SD-WM-DQO-027, Rev. 0B, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Defines the amount, type, and quality of information needed to characterize the residual waste in tank 241-AX-104 in support of the Hanford Tanks Initiative Project.
- Banning, D. L., 1998, Hanford Tank Initiative Tank 241-AX-104 Upper Vadose Zone Demonstration Data Quality Objectives, HNF-2326, Rev. 0, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Evaluates the use of cone penetrometer technologies and provides information supporting vadose zone soils characterization adjacent to tank 241-AX-104.
- Dukelow, G. T., J. W. Hunt, H. Babad, and J. E. Meacham, 1995, *Tank Safety Screening Data Quality Objective*, WHC-SD-WM-SP-004, Rev. 2, Westinghouse Hanford Company, Richland, Washington.
 - Determines whether tanks are under safe operating conditions.
- Meacham, J. E., D. L. Banning, M. R. Allen, and L. D. Muhlestein, 1997, Data Quality Objective to Support Resolution of the Organic Solvent Safety Issue, HNF-SD-WM-DQO-026, Rev. 0, DE&S Hanford, Inc. for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Contains requirements for the organic solvents DOO.

- Osborne, J. W., and L. L. Buckley, 1995, *Data Quality Objectives for Tank Hazardous Vapor Safety Screening*, WHC-SD-WM-DQO-002, Rev. 2, Westinghouse Hanford Company, Richland, Washington.
 - Contains requirements for addressing hazardous vapor issues.
- Schreiber, R. D., 1997, Memorandum of Understanding for the Organic Complexant Safety Issue Data Requirements, HNF-SD-WM-RD-060, Rev. 0, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Contains requirements, methodology and logic for analyses to support organic complexant issue resolution.

II. ANALYTICAL DATA - SAMPLING OF TANK WASTE AND WASTE TYPES

IIa. Sampling of Tank 241-AX-104

: :

- Buckingham, J. S., 1978, *Heat Generation of Residual Sludge in Tank 104 AX*, (letter 60120-78-040 J to C. D. Campbell, June 15), Rockwell Hanford Operations, Richland, Washington.
 - Presents a heat generation estimate based on a sample of the residual tank sludge; however, the specific sampling event is unknown. Also presents the results of an analysis of sludge in tank 004-AR, which contained sluiced 241-AX-104 material. A heat generation rate estimate was derived based on this analytical data.
- Crawford, B. A., 1998, *Tank 241-AX-104 Residual Solids Leach Test Results*, TWR-3548, Rev. 0, Numatec Hanford Corporation for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Presents results from the composite and leach test analyses on the November 1997 auger samples.

- Esch, R. A., 1998, Tank 241-AX-104, Auger Samples, 97-AUG-001, 97-AUG-002, 97-AUG-003, and 97-AUG-004 Analytical Results for the Final Report, HNF-SD-WM-DP-298, Rev. 0, Waste Management Federal Services of Hanford Inc.for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Contains results for the analysis of the individual 1997 auger samples.
- Horton, J. E., and J. S. Buckingham, 1974, *Characterization and Analysis of Tank 104-AX Sludge*, (letter to O. R. H. Rasmussen, October 14), Atlantic Richfield Hanford Company, Richland, Washington.
 - Presents analytical results for a May 1974 sludge sample.
- Koegler, S. S., 1976, A and AX Tank Sludge Heat Generation Rates, (letter to R. E. Felt, February 12), Atlantic Richfield Hanford Company, Richland, Washington.
 - Calculates a heat generation rate based on the ⁹⁰Sr and ¹³⁷Cs data from the May 1974 sludge sample.
- Lockrem, L. L., 1997, Revised Data Tables for Tank Vapor Database on Tanks 241-A-106, 241-AX-104, and 241-TX-106, (letter NHC-9756182 to M. R. Adams, July 17), Numatec Hanford Corporation for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Provides revised data tables from the January 1997 vapor sampling event.
- Starr, J. L., 1977, 104-AX Sludge Analysis, (letter to F. M. Jungfleisch, October 14), Rockwell Hanford Operations, Richland, Washington.
 - Presents analytical results for a September 1977 sludge sample.
- Viswanath, R. S., G. S. Caprio, J. G. Douglas, M. J. Duchsherer, E. S. Mast, L. A. Pingel, M. Stauffer, D. B. Bonfoey, and G. A. Fries, 1998, *Tank Vapor Sampling and Analysis Data Package for Tank 241-AX-104, Sampled January 23, 1997*, HNF-SD-WM-DP-278, Rev. 0C, Numatec Hanford Corporation for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Presents field data and analytical results from the January 23, 1997 headspace vapor sampling of tank 241-AX-104.

- Wheeler, R. E., 1975, *Analysis of Tank Farm Samples*, (letter to R. L. Walser, October 2), Atlantic Richfield Hanford Company, Richland, Washington.
 - Contains results from a 1975 liquid sample.

IIb. Sampling of PUREX High-Level Waste

- Buckingham, J. S., 1978, *Heat Generation of Residual Sludge in Tank 104 AX*, (letter 60120-78-040 J to C. D. Campbell, June 15), Rockwell Hanford Operations, Richland, Washington.
 - Presents a heat generation estimate based on a sample of the residual tank sludge; however, the specific sampling event is unknown. Also presents the results of an analysis of sludge in tank 004-AR, which contained sluiced 241-AX-104 material. A heat generation rate estimate was derived based on this analytical data.
- Van Tuyl, H. H., 1958, Composition of Some PUREX Plant IWW Solutions, HW-57280, General Electric Company, Richland, Washington.
 - Presents compositions of some of the PUREX Plant IWW (now known as P2) solutions.

III. COMBINED ANALYTICAL/NON-ANALYTICAL DATA

IIIa. Inventories from Campaign and Analytical Information

- Agnew, S. F., J. Boyer, R. A. Corbin, T. B. Duran, J. R. Fitzpatrick, K. A. Jurgensen, T. P. Ortiz, and B. L. Young, 1997, *Hanford Tank Chemical and Radionuclide Inventories: HDW Model Rev. 4*, LA-UR-96-3860, Rev. 0, Los Alamos National Laboratory, Los Alamos, New Mexico.
 - Contains waste type summaries and primary chemical compound/analyte and radionuclide estimates for sludge, supernatant, and solids.

- Allen, G. K., 1976, Estimated Inventory of Chemicals Added to Underground Waste Tanks, 1944 1975, ARH-CD-601B, Atlantic Richfield Hanford Company, Richland, Washington.
 - Contains major components for waste types, and some assumptions.
 Purchase records are used to estimate chemical inventories.
- Brevick, C. H., L. A. Gaddis, and E. D. Johnson, 1996, *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Area*, WHC-SD-MW-ER-349, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.
 - Contains summary information from the supporting document as well as in-tank photo collages and the solid composite inventory estimates Rev. 0 and Rev. 0A.

IIIb. Compendium of Data from Other Physical and Chemical Sources

- Brevick, C. H., J. L. Stroup, and J. W. Funk, 1997, Supporting Document for the Historical Tank Content Estimate for AX-Tank Farm, WHC-SD-WM-ER-309, Rev. 1B, Fluor Daniel Northwest for Fluor Daniel Hanford, Inc., Richland, Washington.
 - Contains historical data and solid inventory estimates. The appendices
 contain level history AutoCAD sketches, temperature graphs, surface level
 graphs, cascade/dry well charts, riser configuration drawings and tables,
 in-tank photos, and tank layer model bar charts and spreadsheets.
- Brevick, C. H., L. A. Gaddis, and E. D. Johnson, 1995, *Tank Waste Source Term Inventory Validation, Vol I & II*, WHC-SD-WM-ER-400, Rev. 0, Westinghouse Hanford Company, Richland, Washington.
 - Contains a quick reference to sampling information in spreadsheet or graphical form for 23 chemicals and 11 radionuclides for all the tanks.

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